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Cimigenol-3-*O*-*β*-D-xylopranoside methanol solvate

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The title compound, (23*R*,24*S*)-16b,23:16a,24-diepoxy-15a,25-dihydroxy-9,19-cyclolanostane-3-*O*-*β*-D-xylopyranoside methanol solvate, $C_{35}H_{56}O_9\cdot CH_4O$, is a cycloartane triterpene glycoside which was isolated from the rhizomes of *Actaea asiatica*. The molecule contains five six-membered rings, two five-membered rings and a three-membered ring. The crystal structure is stabilized by intra- and intermolecular O—H···O hydrogen bonds.

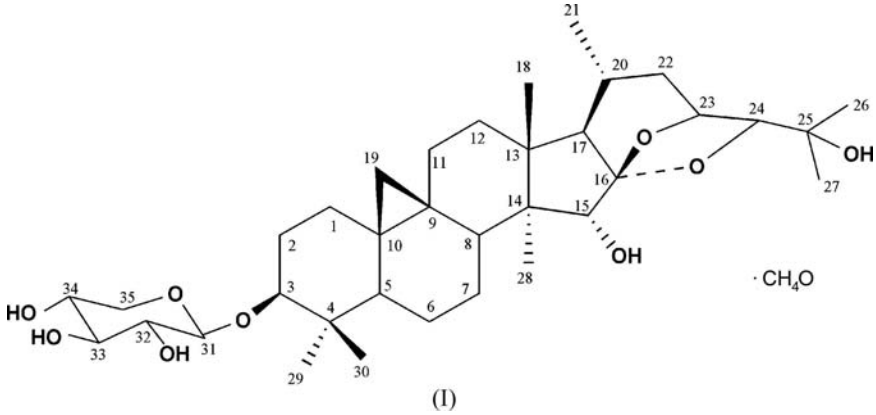
Key indicators

Single-crystal X-ray study
T = 294 K
Mean *σ*(C–C) = 0.007 Å
Disorder in solvent or counterion
R factor = 0.057
wR factor = 0.183
Data-to-parameter ratio = 10.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Actaea asiatica Hara (ranunculaceae) is widely distributed in the southwest and northwest of the People's Republic of China. As a Chinese folk medicine, its rhizomes are used to treat headache, sore throat, measles, pertussis and prolapse of the uterus (Wan, 1990). Our investigation of the bioactive constituents of the rhizomes of *A. actaca* led to the isolation of cimigenol-3-*O*-*β*-D-xylopranoside, (I). The structure of the compound has been elucidated by ^{1}H NMR and ^{13}C NMR spectroscopy (Nobuko *et al.*, 1972; Pan *et al.*, 2002). In order to establish the structure unequivocally, we have carried out a single-crystal X-ray diffraction analysis.



(I)

The molecular structure of (I) is shown in Fig. 1. The molecule contains five six-membered rings (*A*, C1–C5/C10; *B*, C5–C10; *C*, C8/C9/C11–C14; *E*, C16/C17/C20/C22/C23/O3; *H*, C31–C35/O9) and two five-membered rings (*D*, C13–C17; *F*, C16/O3/C23/C24/O4). Rings *A* and *H* adopt chair conformations, while ring *B* adopts a slightly distorted half-chair conformation. Ring *C* adopts a distorted boat conformation and ring *E* adopts a boat conformation. Rings *D* and *F* each exhibit an envelope conformation. All rings are *trans* fused. Atom C9 belongs to two six-membered rings and has a distorted tetrahedral bonding geometry, with the C19—C9—C10 [59.5 (3)°] and C19—C9—C8 [113.8 (4)°] angles deviating

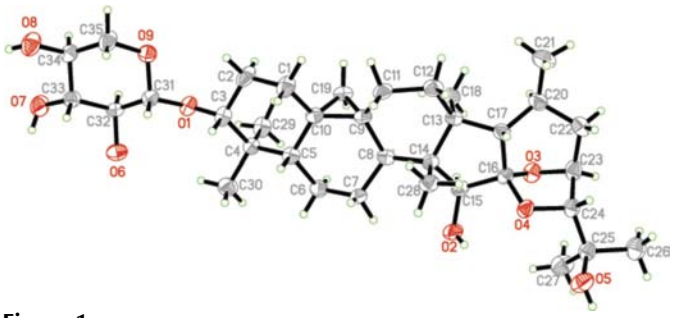


Figure 1
The molecular structure of (I), showing the atom-labelling scheme and displacement ellipsoids drawn at the 30% probability level. The disordered methanol solvent has been omitted.

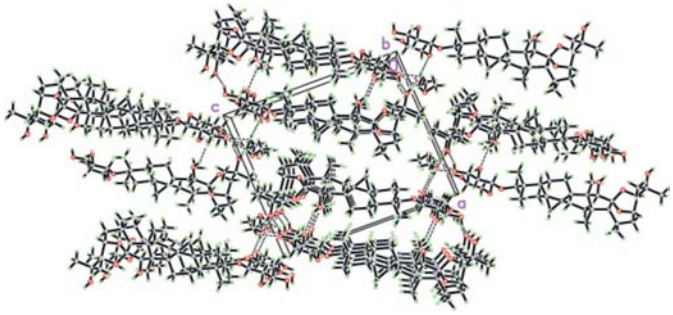


Figure 2
Part of the crystal structure of (I) with hydrogen bonds shown as dashed lines. The disordered methanol solvent has been omitted.

from the ideal tetrahedral values. A similar geometric distortion is observed in the environment of atom C10, *viz.* C19—C10—C9 = 59.1 (3)° and C19—C10—C5 = 123.6 (4)°. In the crystal structure, the packing of the molecules is stabilized by intra- and intermolecular O—H···O hydrogen bonds (Table 1 and Fig. 2).

Experimental

The rhizomes of *A. asiatica* were collected in August 2004 in Hefeng, Hubei province, and were identified by Professor Ding-Rong Wan (School of Life Sciences, South-Central University for Nationalities). A voucher specimen (D20040901) was deposited at the School of Pharmacy, Tianjin Medical University, China. The rhizomes of *A. asiatica* (2.6 kg) were refluxed three times with 95% EtOH (5000 ml each) for 5 h. The extract was concentrated *in vacuo* to give a residue (600 g), which was suspended in water, and then partitioned with petroleum ether (PE), EtOAc and *n*-BuOH, successively. The EtOAc extract (220 g) was chromatographed on a silica gel column, eluted with solvents of increasing polarity to give 14 fractions. Fraction 5 (2.1 g) was separated by flash chromatography on a column (C_{18}) and then purified by pre. HPLC-ODS to give (I) (50 mg). Suitable crystals were obtained by slow evaporation of a methanol solution at room temperature. ^{13}C NMR (75 MHz, C_5ND_5, p.p.m.): 32.9 (C1), 30.5 (C2), 89.0 (C3), 41.8 (C4), 48.1 (C5), 21.8 (C6), 26.8 (C7), 49.0 (C8), 20.5 (C9), 27.1 (C10), 26.9 (C11), 34.6 (C12), 42.4 (C13), 47.8 (C14), 80.6 (C15), 112.4 (C16), 60.1 (C17), 20.0 (C18), 31.3 (C19), 24.5 (C20), 19.9 (C21), 38.5 (C22), 72.3 (C23), 90.6 (C24), 71.3 (C25), 25.9 (C26), 26.2 (C27), 12.2 (C28), 27.6 (C29), 15.8 (C30), 107.8 (C31), 75.7 (C32), 78.8 (C33), 71.6 (C34), 67.5 (C35).

Crystal data

$C_{35}H_{56}O_9\cdot CH_4O$
M_r = 652.84
Monoclinic, $P2_1$
a = 14.500 (3) Å
b = 6.9301 (14) Å
c = 17.267 (4) Å
β = 93.352 (4)°
V = 1732.1 (6) Å^3
Z = 2
D_x = 1.252 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 294 (2) K
Block, colourless
0.22 × 0.18 × 0.10 mm

Data collection

Bruker SMART 1000 diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.981, T_{max} = 0.991
10507 measured reflections
4482 independent reflections
2630 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 28.3°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.183
S = 1.06
4482 reflections
449 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0865P)^2 + 0.4276P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O6^{i}	0.82	2.19	2.880 (5)	143
O2—H2···O4	0.82	2.32	2.726 (5)	111
O5—H5···O10^{ii}	0.82	2.02	2.841 (7)	175
O6—H6···O8^{iii}	0.82	2.11	2.916 (6)	168
O7—H7···O5^{iv}	0.82	2.01	2.727 (6)	146
O8—H8···O10^{v}	0.82	2.07	2.846 (6)	159
O10—H10···O9^{vi}	0.85	2.02	2.867 (6)	180
O10′—H10′···O7^{vii}	0.82	2.40	3.087 (13)	142

Symmetry codes: (i) $-x, y+\frac{1}{2}, -z+1$; (ii) $-x+1, y+\frac{3}{2}, -z$; (iii) $x, y+1, z$; (iv) $-x, y-\frac{1}{2}, -z+1$; (v) $x-1, y, z+1$; (vi) $-x+1, y-\frac{1}{2}, -z+1$; (vii) $x+1, y, z-1$.

In the absence of significant anomalous dispersion effects, Friedel pairs were merged and the absolute configuration was assigned as in the publications of Nobuko *et al.* (1972) and Pan *et al.* (2002). All H atoms were positioned geometrically (C—H = 0.96–0.98 Å and O—H = 0.82 Å) and refined in the riding-model approximation. For the CH and CH_2 groups, U_{iso}(H) = 1.2U_{eq}(C), and for methyl and OH groups, U_{iso}(H) = 1.5U_{eq}(C,O). The methanol solvent molecule is disordered over two sites with refined occupancies of 0.691 (7):0.309 (7).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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